इंटरनेट मानक

Disclosure to Promote the Right To Information

Whereas the Parliament of India has set out to provide a practical regime of right to information for citizens to secure access to information under the control of public authorities, in order to promote transparency and accountability in the working of every public authority, and whereas the attached publication of the Bureau of Indian Standards is of particular interest to the public, particularly disadvantaged communities and those engaged in the pursuit of education and knowledge, the attached public safety standard is made available to promote the timely dissemination of this information in an accurate manner to the public.

"जानने का अधिकार, जीने का अधिकार"
Mazdoor Kisan Shakti Sangathan
"The Right to Information, The Right to Live"

"पुराने को छोड नये के तरफ"
Jawaharlal Nehru
"Step Out From the Old to the New"

IS 10977 (1984): Pentaerythritol for explosive industry
[CHD 26: Explosives and Pyrotechnics]

"ज्ञान से एक नये भारत का निर्माण"
Satyanarayan Gangaram Pitroda
"Invent a New India Using Knowledge"

Act Number 22 of 2005
The Indian Parliament
RIGHT TO INFORMATION

Made Available By
Public.Resource.Org

"ज्ञान एक ऐसा खजाना है जो कभी चुराया नहीं जा सकता है"
Bhartṛhari—Nītiśatakam
"Knowledge is such a treasure which cannot be stolen"

BLANK PAGE

BRUTUM FULMEN
IGNORANTIA
JURIS
NON EXCUSAT
PROTECTED BY COPYRIGHT

IS : 10977 - 1984

Indian Standard

SPECIFICATION FOR PENTAERYTHRITOL FOR EXPLOSIVE INDUSTRY

UDC 661·7 : 547·427·1 : 662·1 ·4

© *Copyright* 1984

INDIAN STANDARDS INSTITUTION
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

Gr 5 *November* 1984

AMENDMENT NO. 1 AUGUST 1990
TO
IS 10977:1984 SPECIFICATION FOR PENTAERYTHRITOL FOR EXPLOSIVE INDUSTRY

[*Page* 4, *clause* 3.3(a)] - Delete '*Max*'.

(CHD 26)

Reprography Unit, BIS, New Delhi, India

Indian Standard

SPECIFICATION FOR PENTAERYTHRITOL FOR EXPLOSIVE INDUSTRY

Explosives and Pyrotechnics Sectional Committee, CDC 51

Chairman	*Representing*
Shri R. G. Deolaliker	Directorate General Ordnance Factories, Calcutta
Members	
Shri K. P. R. Pillay (*Alternate* to Shri R. G. Deolaliker)	
Shri Avtar Singh	National Fertilizers Limited, New Delhi
Shri P. K. Banerjee	National Mineral Development Corporation Ltd, Hyderabad
Shri S. D. Garg (*Alternate*)	
Shri B. R. Dave	Chief Controller of Explosives, Nagpur
Shri H. Mukherjee (*Alternate*)	
Shri C. M. Deosthale	IBP Co Ltd, New Delhi
Dr P. R. Nambiar (*Alternate*)	
Director (Mines Safety Equipment)	Directorate General of Mines Safety, Dhanbad
Joint Director of Mines Safety (*Alternate*)	
Shri A. Grahadurai	National Fireworks Factory, Sivakasi
Shri S. Ramesh Babu (*Alternate*)	
Shri J. W. Kawathekar	Narendra Explosives Ltd, Dehra Dun
Shri V. Krishnamurthi	Central Mining Research Station, Dhanbad
Shri M. M. Bhattacharya (*Alternate*)	
Shri B. Minj	Directorate General of Technical Development, New Delhi
Shri A. K. Chatterjee (*Alternate*)	
Dr S. B. Ray	Ministry of Defence (DGI)
Shri G. D. Palkar (*Alternate*)	
Shri P. R. Roy	Central Mine Planning & Design Institute Limited, Ranchi
Dr N. Sadasivan	Ministry of Defence (R & D)
Shri K. U. B. Rao (*Alternate*)	
Shri S. S. Salodkar	Coal India Ltd, Calcutta
Shri U. W. Datey (*Alternate*)	

(*Continued on page* 2)

© *Copyright* 1984
INDIAN STANDARDS INSTITUTION
This publication is protected under the *Indian Copyright Act* (XIV of 1957) and reproduction in whole or in part by any means except with written permission of the publisher shall be deemed to be an infringement of copyright under the said Act.

(*Continued from page* 1)

Members	*Representing*
Shri P. Satyanarayana	Development Commissioner (Small Scale Industries), New Delhi
Dr S. K. Kapoor (*Alternate*)	
Shri S. Seshan	Indian Explosives Ltd, Calcutta
Shri R. K. Guha (*Alternate*)	
Dr S. R. Yoganarasimhan	IDL Chemicals Ltd, Hyderabad
Dr G. D. Prasad (*Alternate*)	
Shri S. K. Mathur, Director (Chem) (*Secretary*)	Director General, ISI (*Ex-officio Member*)

Raw Materials Subcommittee, CDC 51 : 4

Convener	
Shri M. G. Bhagwat	Ministry of Defence (DGI)
Members	
Shri N. V. Pillai (*Alternate* to Shri M. G. Bhagwat)	
Dr N. S. Bankar	Explosives Research and Development Laboratory, Pune
Shri K. M. Agarwal (*Alternate*)	
Shri A. V. Chandorkar	IBP Co Ltd, New Delhi
Dr P. R. Nambiar (*Alternate*)	
Shri A. Grahadurai	National Fireworks Factory, Sivakasi
Shri A. P. Manjrekar	IDL Chemicals Ltd, Hyderabad
Shri R. Srinivasan (*Alternate*)	
Dr Anil Pandit	Deepak Nitrite Ltd, Vadodara
Shri J. T. Vora (*Alternate*)	
Shri K. P. R. Pillay	Directorate General Ordnance Factories, Calcutta
Shri K. Nanda Kumar (*Alternate*)	
Shri N. K. Samanta	Indian Explosives Ltd, Calcutta
Shri B. S. Roy (*Alternate*)	

Indian Standard

SPECIFICATION FOR PENTAERYTHRITOL FOR EXPLOSIVE INDUSTRY

0. FOREWORD

0.1 This Indian Standard was adopted by the Indian Standards Institution on 30 March 1984, after the draft finalized by the Explosives and Pyrotechnics Sectional Committee had been approved by the Chemical Division Council.

0.2 Indian Standard specification for pentaerythritol (IS : 7619-1974) covers the requirements of the material for use in the manufacture of paint coatings and printing inks. It does not cover nitration grade of the material required by explosive industry. This standard is intended to cover the material used for the production of pentaerythritol tetranitrate for use in explosive and pyrotechnic compositions.

0.3 It is advisable for the users to carry out trial nitration test with the material after ensuring conformity to this specification to assess the desired quality of the end product.

0.4 For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS : 2-1960*. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

1. SCOPE

1.1 This standard prescribes requirements and the methods of sampling and test for pentaerythritol, nitration grade, for explosive industry.

2. GRADES

2.1 The material shall be of two grades, namely, Grade 1 and Grade 2.

---

*Rules for rounding off numerical values (*revised*).

3. REQUIREMENTS

3.1 The material shall be in the form of white crystalline powder, free from impurities visible to the naked eye.

3.2 Flow Properties — The material shall flow without interruption from a glass funnel with a 5-mm bore and 60° angle.

3.3 Particle Size — The particle size of the material shall be as follows:

a) Passing through 1·00-mm IS Sieve, percent by mass, *Max*	100
b) Passing through 212-micron IS Sieve, percent by mass, *Max*	1

3.4 The material shall also comply with the requirements laid down in Table 1 when tested according to the methods prescribed in Appendix A. Reference to the relevant clauses of Appendix A is given in col 5 of the table.

4. PACKING AND MARKING

4.1 Packing — The material shall be packed in polyethylene-lined jute or paper bags or drums as agreed to between the purchaser and the supplier.

4.2 Marking — Each container shall be securely closed and shall bear legibly and indelibly the following information:

a) Name and grade of the material;

b) Net mass;

c) Month and year of manufacture;

d) Name of the manufacturer and/or his trade-mark, if any; and

e) Lot number in code or otherwise to enable the batch of manufacture to be traced from records.

4.2.1 The containers may also be marked with the ISI Certification Mark.

NOTE — The use of the ISI Certification Mark is governed by the provisions of the Indian Standards Institution (Certification Marks) Act and the Rules and Regulations made thereunder. The ISI Mark on products covered by an Indian Standard conveys the assurance that they have been produced to comply with the requirements of that standard under a well-defined system of inspection, testing and quality control which is devised and supervised by ISI and operated by the producer. ISI marked products are also continuously checked by ISI for conformity to that standard as a further safeguard. Details of conditions under which a licence for the use of the ISI Certification Mark may be granted to manufacturers or processors, may be obtained from the Indian Standards Institution.

TABLE 1 REQUIREMENTS FOR PENTAERYTHRITOL FOR EXPLOSIVE INDUSTRY

(*Clause* 3.4)

Sl No.	Characteristic	Requirement		Method of Test (Ref to Cl No. in Appendix A)
		Grade 1	Grade 2	
(1)	(2)	(3)	(4)	(5)
i)	Moisture, percent by mass, *Max*	0·1	0·1	A-3
ii)	Melting point,°C	252 to 260	249 to 255	A-4
iii)	Pentaerythritol (mono PE content), percent by mass, *Min*	99·2	97·5 to 98·5	A-5
iv)	Hydroxyl value, percent by mass, *Min*	49·5	49·0	A-6
v)	Di Penta content, percent by mass, *Max*	0·4	0·9	A-7
vi)	PE formals, percent by mass, *Max*	0·4	0·5	
vii)	Sulphated ash, percent by mass, *Max*	0·05	0·1	A-8
viii)	Water insolubles, percent by mass, *Max*	0·1	0·1	A-9
ix)	Chlorides (as Cl), percent by mass, *Max*	0·01	0·01	A-10
x)	Formic acid, percent by mass, *Max*	0·005	0·005	A-11
xi)	Iron (as Fe), percent by mass, *Max*	0·002	0·002	A-12

5. SAMPLING

5.1 Representative samples of the material shall be drawn and their conformity to the standard determined as prescribed in Appendix B.

APPENDIX A

(*Clause* 3.4)

METHODS OF TEST FOR PENTAERYTHRITOL FOR EXPLOSIVE INDUSTRY

A-1. QUALITY OF REAGENTS

A-1.1 Unless specified otherwise, pure chemicals and distilled water (*see* IS : 1070-1977*) shall be employed in test.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

A-2. PREPARATION OF SAMPLE

A-2.1 Weigh about 100 g of the material and dry for 30 minutes in an oven at a temperature of 110 ± 2°C. Cool in a desiccator and weigh. Dry again and repeat until constant mass is attained.

A-3. DETERMINATION OF MOISTURE

A-3.1 Procedure — Weigh accurately about 1·0 g of the sample into a weighing bottle which has been dried, cooled in the desiccator and then weighed. Place the weighing bottle in the air oven for about 1 hour at 100 ± 1°C. Remove the weighing bottle from the oven, cool in a desiccator to room temperature and weigh. Repeat this procedure. Keep the weighing bottle in the oven only for half an hour each time until the difference between two successive weighings is less than 1 mg.

A-3.2 Calculation

$$\text{Moisture content, percent by mass} = \frac{100 \times M_1}{M}$$

where

M_1 = loss in mass in g of the sample upon drying, and
M = mass in g of the sample taken for the test.

A-4. DETERMINATION OF MELTING POINT

A-4.1 Procedure — Carry out the determination of melting point according to the method prescribed in IS : 5762-1970†.

---

*Specification for water for general laboratory use (*second revision*).

†Methods for determination of melting point and melting range.

A-5. DETERMINATION OF PENTAERYTHRITOL (MONO PE CONTENT)

A-5.0 Outline of the Method — Pentaerythritol reacts with benzaldehyde in the presence of strong acid to form dibenzylacetal.

$$C(\ CH_2OH\ )_4 + 2C_6H_5CHO \rightarrow C_6H_5CH\ (\ OCH_2\ )_2\ C\ (\ OCH_2\ )_2\ CHC_6H_5 + 2H_2O$$

The dibenzylacetal so formed is filtered off, washed and dried to constant mass. A correction is made for the slight solubility of dibenzylacetal under the conditions of the test. Dipentaerythritol and polypentaerythritols which may be present in the sample do not react.

A-5.1 Reagents

A-5.1.1 *Concentrated Hydrochloric Acid*

A-5.1.2 *Benzaldehyde Reagent* — Mix 15 ml of benzaldehyde with 100 ml of anhydrous methanol. The benzaldehyde bottle is sealed immediately to avoid oxidation to benzoic acid.

A-5.1.3 *Wash Solution* — Mix methanol and water in 1 : 1 ratio and cool in ice.

A-5.2 Procedure — Weigh 0·5 g of the prepared sample into a dry 250-ml conical flask. Add 5 ml of water. Heat it slightly to dissolve the sample. While hot, add 20 ml of the benzaldehyde followed by the addition of 12 ml of concentrated hydrochloric acid. Stopper the flask with a rubber cork and allow to stand for 30 minutes. Swirl the contents of the flask occasionally. Then keep the flask in ice for 1 hour with occasional swirling. Filter the solution through a dried and weighed G4 sintered crucible. Wash precipitate with 6-10 ml portions of ice cold wash solution. Totally 40 ml of ice cold wash solution is used for complete transfer and washing of the precipitate. Dry the crucible with the precipitate at 100°C and weigh to a constant mass.

A-5.3 Calculation

$$\text{Pentaerythritol (mono PE content), percent by mass} = \frac{(\ M_1 + 0{\cdot}03\ ) \times 43{\cdot}59}{M_2}$$

where

M_1 = mass in g of the precipitate obtained, and

M_2 = mass in g of the material taken for the test.

NOTE 1 — 0·03 is the correction taken for calculation as the precipitate is soluble in methanol/water mixture at the cold temperature.

NOTE 2 — The benzaldehyde method of determination of purity of PE gives a slightly higher value as the PE formals also get precipitated along with the mono PE in the benzaldehyde complex. Hence, a correction has to be made for the PE formals by subtracting the value for PE formals obtained by the GLC method.

A-6. DETERMINATION OF HYDROXYL VALUE

A-6.0 Outline of the Method — The material is acetylated with acetic anhydride. The excess of anhydride is titrated back after hydration with caustic soda.

A-6.1 Reagents

A-6.1.1 *Acetic Anhydride*

A-6.1.2 *Pyridine*

A-6.1.3 *Potassium Hydroxide*

A-6.1.4 *Sodium Hydroxide Solution* — 1 N.

A-6.1.5 *Acetic Anhydride — Pyridine Solution* — 2·5 N in pyridine prepared as follows:

> 29·6 ml of acetic anhydride made up to 250 ml with pure pyridine is dried over potassium hydroxide for 16 hours. The mixture is agitated and kept in the dark in a glass-stoppered bottle. This reagent has a yellow colour, which tends to decrease with time. (If the colour turns darker, or if the reagent is older than one week, it should not be used).

A-6.1.6 *Phenolphthalein Indicator Solution* — 0·5 percent (*m/v*). Dissolve 0·5 g of phenolphthalein in 100 ml of rectified spirit and make it faintly pink by the addition of dilute ammonium hydroxide.

A-6.2 Procedure — Place 0·5 to 0·6 g of the prepared sample in the acetylation flasks and add 25 ml of acetic anhydride-pyridine solution with a burette. Place air-cooled condenser on top and heat the flask in the glycerine bath at 150°C for 30 minutes. (They should not be heated any longer, otherwise the results will be too low).

Remove the flasks from the bath, rinse the condenser with 30 to 50 ml of water and cool the flasks for 20 minutes under a jet of water.

When the temperature has reached 20°C, titrate the contents of the flasks with 1 N sodium hydroxide solution using phenolphthalein as indicator. The titrating solution shall be added at a rate of 15-20 drops per minute. The last 10 ml shall be added very slowly under good shaking. Carry out blank test on 25 ml of acetic anhydride-pyridine solution.

A-6.3 Calculation

$$\text{Hydroxyl value, percent by mass} = \frac{1{\cdot}70\,(V_1 - V_2) \times N}{M}$$

where

V_1 = volume in ml of standard sodium hydroxide solution used in the blank,

V_2 = volume in ml of standard sodium hydroxide solution used in the titration,

N = normality of sodium hydroxide solution, and

M = mass in g of the sample taken for the test.

A-7. DETERMINATION OF DIPENTA AND PE FORMALS

A-7.0 Principle — Finely ground pentaerythritol (PE) is treated with hexamethyl disilazane (HMDS) and trimethyl chlorosilane (TMCS). The volatile TMCS ethers of PE's are produced which are injected into gas-liquid chromatograph for separation. The chromatograms are used for finding the percentage of each component from the peak areas.

A-7.1 Preparation of Silyl Ethers

A-7.1.1 *Method I* — 20 mg of pentaerythritol is taken and 0·5 ml of pyridine is added and heated to 70°C in a water-bath with stirring. To this stirred solution, 0·5 ml of hexa methyl disilazane is added. The mixture is then heated for 15 minutes at 70°C with thorough stirring. The contents are then cooled to 30°C and 0·8 ml of trimethyl chlorosilane is added. The contents are again heated with brisk stirring. The reaction is complete at the end of 3 hours at 70°C. The solution is allowed to cool and then centrifuged. The supernatant liquid is the volatile silyl ethers of PE's and other compounds. These ethers are quantitatively produced under the conditions mentioned above and they are ready for injecting into the gas-liquid chromatograph.

A-7.1.2 *Method II* – 20 ml of pentaerythritol is mixed with 30 mg of anhydrous sodium sulphate. To this mixture, 1·5 ml of HMDS is added and heated for 30 minutes at 70°C in a water-bath. There shall be continuous stirring. Hydrochloric acid gas is passed into the mixture for a minute and stirred briskly. The gas is generated either by heating concentrated sulphuric acid-hydrochloric acid mixture or by the addition of concentrated sulphuric acid to sodium chloride This is done in a 50-ml conical flask with a two holes rubber stopper, one carrying a small thistle funnel and the other a bent tube for hydrochloric acid gas delivery. The mixture is heated for 3 hours at 70°C so that silylation is complete. The mixture is cooled and centrifuged. The TMS ethers are ready for gas chromatographic analysis.

A-7.2 Reagents

A-7.2.1 *Pyridine* — Dried over potassium hydroxide flakes.

A-7.2.2 *Hexamethyl Disilazane*

A-7.2.3 *Trimethyl Chloro Silane*

A-7.2.4 *Sodium Sulphate* — anhydrous.

A-7.2.5 *Hydrochloric Acid* — gas.

A-7.3 Column Details

Column	15 cm long, 3 mm OD stainless steel column
Coating agent	2 percent silicone
Detector	Flame ionisation detector
Carrier gas	Nitrogen
Sample size	2 μl
Mode of operation	Programmed heating
Initial temperature, °C	100
Final temperature, °C	250
Initial time, minutes	3
Rate of heating, °C/min	8
Final time, minutes	8
Injection port temperature, °C	300
Manifold temperature, °C	300
Attenuation for PE	X 32
For others	X 2
Range	X 100
Order of elution	Solvent, mono-PE, di-PE, bisformal and tri-PE
Solvent used	Pyridine
Standard sample	
Solid support	Gas chrom — p 100/120 mesh

A-7.4 Determination of Calibration Factors

A-7.4.1 The standard samples are silylated as per procedure and the silyl ethers are injected into the GLC column as described in **A-7.3**. The peak areas are calculated. The areas are divided by the known percentages and calibration factor determined as follows:

Area of the peak PE	A
Area of the peak di-PE	B
Area of the peak bisformal	C
Area of the peak tri-PE	D

A-7.4.2 *Percentage in the Sample*

PE	$98/A$	(A)
Di-PE	1·0	
Bisformal	0·5	
Tri-PE	0·5	

A-7.4.3 Calibration factors are:

PE	98	(A')
Di-PE	$\frac{1\cdot0}{B}$	(B')
Bisformal	$\frac{0\cdot5}{C}$	(C')
Tri-PE	$\frac{0\cdot5}{D}$	(D')

NOTE — Run the standard samples whenever a new set of sample is to be analyzed.

A-7.5 Determination of Percentage in the Sample of PE

A-7.5.1 The sample is silylated as per procedure described. The silyl ether is injected into the column as described in **A-7.3**. The peak areas are calculated and the percentages determined as follows:

Peak area of PE = E

Peak area of di-PE = F

Peak area of bisformal = G

Peak area of tri-PE = H

Mass unit of PE = $E \times A' = E'$

Mass unit of di-PE = $F \times B' = F'$

Mass unit of bisformal = $G \times C' = G'$

Mass unit of tri-PE = $H \times D' = H'$

Let $E' + F' + G' + H'$ = K

PE, percent by mass = $\frac{(E')}{(K)} \times 100$

$$\text{Di-PE, percent by mass} = \frac{F'}{K} \times 100$$

$$\text{Bisformal, percent by mass} = \frac{G'}{K} \times 100$$

$$\text{Tri-PE, percent by mass} = \frac{H'}{K} \times 100$$

A-8. DETERMINATION OF SULPHATED ASH

A-8.1 Procedure — Weigh accurately about 3 g of the material and cautiously evaporate in a tared procelain crucible of 45 mm diameter and 40 mm high, over a gentle flame. Calcine the residue by adding a drop of concentrated sulphuric acid for 15 minutes at 800 to 1 000°C and weigh the crucible till constant mass is obtained.

A-8.2 Calculation

$$\text{Sulphated ash, percent by mass} = \frac{M_1}{M} \times 100$$

where

M_1 = mass in g of the residue obtained after ignition, and
M = mass in g of the material taken for the test.

A-9. DETERMINATION OF WATER INSOLUBLES

A-9.1 Procedure — Weigh 10 g of dry pentaerythritol and dissolve in 100 ml of warm water. Filter through a tared G 4 sintered glass filter funnel. Wash with water, dry for 2 hours at 70°C and weigh to a constant mass.

A-9.2 Calculation

$$\text{Water insoluble residue, percent by mass} = \frac{M_1 - M_2}{M} \times 100$$

where

M_1 = mass in g of filter funnel after the filtration,
M_2 = tare in g of filter funnel, and
M = mass in g of the material taken for the test.

A-10. DETERMINATION OF CHLORIDE

A-10.1 Principle — Chloride is completely precipitated as silver chloride by the addition of an excess of standard silver nitrate solution and the excess is back titrated with ammonium sulphocyanide solution.

A-10.2 Reagents

A-10.2.1 *Silver Nitrate Solution* — 0·1 N.

A-10.2.2 *Ammonium Sulphocyanide* — 0·1 N.

A-10.2.3 *Concentrated Nitric Acid*

A-10.2.4 *Ferric Alum Indicator* — 40 percent.

A-10.3 Procedure — Weigh about 10 g of dry pentaerythritol accurately in a 250-ml conical flask. Dissolve with heating in 100 ml of water. Prepare a blank by taking 100 ml water in another 250-ml conical flask. Add to each flask 2 ml of concentrated nitric acid followed by 250 ml 0·1 N silver nitrate solution through a pipette. The solutions are titrated against 0·1 N ammonium sulphocyanide using 2 ml of ferric alum indicator till there is a persistent colour change.

A-10.4 Calculation

$$\text{Chloride, as percent by mass} = (A - B) \times N \times \frac{35{\cdot}45}{1\,000} \times \frac{100}{M}$$

where

A = volume of ammonium sulphocyanide consumed for blank titration,

B = volume of ammonium sulphocyanide consumed for sample titration,

N = normality of ammonium sulphocyanide solution, and

M = mass in g of sample taken for the test.

A-11. DETERMINATION OF FORMIC ACID

A-11.1 Reagent

A-11.1.1 *Sodium Hydroxide Solution* — 0·02 N.

A-11.2 Procedure — Weigh about 10 g of dry pentaerythritol accurately in a 200-ml beaker and add 100 ml of boiled water. After cooling titrate with sodium hydroxide solution using phenolphthalein as indicator. The acidity is expressed as formic acid

A-11.3 Calculation

$$\text{Formic acid, percent by mass} = \frac{AN \times 46}{100} \times \frac{1\,000}{M}$$

where

A = volume of sodium hydroxide solution consumed,

N = normality of sodium hydroxide solution, and

M = mass in g of the material taken for the test.

A-12. DETERMINATION OF IRON

A-12.1 Principle — Pentaerythritol is incinerated and the iron content estimated in the ash colorimetrically.

A-12.2 Apparatus

A-12.2.1 *Nessler Cylinder* — 50 ml capacity.

A-12.3 Reagents

A-12.3.1 *Standard Iron Solution* — 0·863 5 g of ferrous ammonium sulphate [$NH_4Fe(SO_4)_2 \cdot 12H_2O$] is dissolved in 100 ml of water and made up to 1 litre (1 ml = 0·1 mg of Fe).

A-12.3.2 *Ammonium Thiocynate Solution* — 4 N.

A-12.3.3 *Dilute Hydrochloric Acid* — 1 : 1.

A-12.4 Procedure — Weigh accurately about 5 g of the material in a previously weighed procelain crucible. Incinerate slowly at a low temperature and then for 1 hour at 800 to 900°C. Cool the crucible and weigh to obtain the ash content. Dissolve the ash in 5 ml of 1 : 1 hydrochloric acid and filter, if necessary, through Whatman No. 42 filter paper and wash the residue with water. Collect the filtrate and washings in a Nessler cylinder. A reference solution is made in another Nessler cylinder comprising 5 ml of 1 : 1 hydrochloric acid, 2 ml of 4 N ammonium sulphocyanide solution and the volume made up to 50 ml with water. Similarly to the sample solution, 2 ml of 4 N ammonium sulphocyanide is added and the volume made up to 50 ml with water. Standard iron solution is added to the reference cylinder dropwise with shaking till the colour matches with that developed in the sample cylinder. Note the volume of standard iron solution consumed.

A-12.5 Calculation

$$\text{Iron, percent by mass} = \frac{100}{M} \times \frac{0 \cdot 1}{1\,000} \times V$$

where

V = volume of standard iron solution, and

M = mass in g of the material taken for the test.

APPENDIX B

(*Clause* 5.1)

SAMPLING OF PENTAERYTHRITOL

B-1. GENERAL REQUIREMENTS OF SAMPLING

B-1.0 In drawing, preparing, storing and handling test samples, the following precautions and directions shall be observed.

B-1.1 Samples shall not be taken in an exposed place.

B-1.2 The sampling instrument shall be clean and dry.

B-1.3 Precautions shall be taken to protect the samples, the material being sampled, the sampling instrument and the containers for samples from adventitious contamination.

B-1.4 To draw a representative sample, the contents of each container selected for sampling shall be mixed as thoroughly as possible by suitable means.

B-1.5 The samples shall be placed in suitable clean, dry and air-tight containers on which the material has no action.

B-1.6 The sample containers shall be of such a size that they are almost but not completely filled by the sample.

B-1.7 Each sample container shall be sealed air-tight after filling and marked with full identification particulars, such as sample number, the date of sampling, batch of manufacture of the material and other important particulars of the consignment.

B-1.8 Samples shall be stored in such a manner that the temperature of the material does not vary unduly from the normal temperature.

B-2. SCALE OF SAMPLING

B-2.1 Lot — In a single consignment all packages of the same size, containing material from the same batch of manufacture, shall constitute a lot. If a consignment is known to consist of packages belonging to different batches of manufacture, packages belonging to the same batch of manufacture shall be grouped together and each such group shall constitute a lot.

B-2.2 For ascertaining the conformity of the material in a lot to the requirements of this specification, samples shall be tested on each lot separately.

B-2.3 The number of packages to be sampled for this purpose shall depend on the size of the lot and shall be in accordance with col 1 and 2 of Table 2.

TABLE 2 SCALE OF SAMPLING

Lot Size N (1)	Number of Containers to be Selected n (2)
Up to 50	3
51 „ 100	4
101 „ 150	5
151 „ 300	6
301 „ 500	7
501 „ 800	8
801 „ 1 300	9
1 301 and above	10

B-2.3.1 These packages shall be selected at random. In order to ensure the randomness of selection, procedures given in IS : 4905-1968* may be followed.

B-3. PREPARATION OF SAMPLES

B-3.1 From each of the packages selected, draw with an appropriate sampling instrument small quantity of material from different portions of the package and mix thoroughly. This shall constitute the representative sample for the package.

B-3.2 From the samples representing different packages selected in **B-2.3**, small but approximately equal quantity of material shall be taken and mixed thoroughly to form a composite sample which shall be sufficient for carrying out triplicate tests for the characteristics given in Table 1. The composite sample shall be divided into three equal parts, one for the purchaser, another for the supplier and the third for the referee.

B-3.3 The referee sample shall bear the seals of both the purchaser and the supplier and shall be kept at a place agreed to between the two. It shall be used in case of any dispute between the two.

B-3.4 The composite samples shall be transferred to separate containers. These sample containers shall then be sealed with stoppers and labelled with full identification particulars given in **B-1.7**.

---

*Methods for random sampling.

B-4. NUMBER OF TESTS

B-4.1 Tests for all characteristics given in Table 1 shall be conducted on the composite sample.

B-5. CRITERIA FOR CONFORMITY

B-5.1 The lot shall be declared as conforming to this specification, if the test results on the composite sample for each of the characteristics meet relevant requirements given in Table 1.

INTERNATIONAL SYSTEM OF UNITS (SI UNITS)

Base Units

QUANTITY	UNIT	SYMBOL
Length	metre	m
Mass	kilogram	kg
Time	second	s
Electric current	ampere	A
Thermodynamic temperature	kelvin	K
Luminous intensity	candela	cd
Amount of substance	mole	mol

Supplementary Units

QUANTITY	UNIT	SYMBOL
Plane angle	radian	rad
Solid angle	steradian	sr

Derived Units

QUANTITY	UNIT	SYMBOL	DEFINITION
Force	newton	N	1 N = 1 kg.m/s^2
Energy	joule	J	1 J = 1 N.m
Power	watt	W	1 W = 1 J/s
Flux	weber	Wb	1 Wb = 1 V.s
Flux density	tesla	T	1 T = 1 Wb/m^2
Frequency	hertz	Hz	1 Hz = 1 c/s (s^{-1})
Electric conductance	siemens	S	1 S = 1 A/V
Electromotive force	volt	V	1 V = 1 W/A
Pressure, stress	pascal	Pa	1 Pa = 1 N/m^2

INDIAN STANDARDS INSTITUTION

Headquarters

Manak Bhavan 9 Bahadur Shah Zafar Marg, NEW DELHI 110002
Telephones 26 60 21 27 01 31

Telegrams: Manaksanstha
(Common to all Offices)

Regional Offices	*Telephone*
*Western : Manakalaya, E9 MIDC, Marol Andheri (East) BOMBAY 400093	6 32 92 95
†Eastern : 1/14 C. I. T Scheme VII M, V I. P. Road, Maniktola, CALCUTTA 700054	36 24 99
Southern C. I T Campus MADRAS 600113	41 24 42
Northern . B69 Phase VII Industrial Focal Point S A S NAGAR 160051 (Punjab)	8 73 28

Branch Offices

Pushpak', Nurmohamed Shaikh Marg Khanpur AHMADABAD 380001	2 63 48 2 63 49
'F' Block, Unity Bldg, Narasimharaja Square, BANGALORE 560002	22 48 05
Gangotri Complex, Bhadbhada Road T. T Nagar, BHOPAL 462003	6 27 16
22E Kalpana Area BHUBANESHWAR 751014	5 36 27
5-8-56C L. N. Gupta Marg, HYDERABAD 500001	22 10 83
R14 Yudhister Marg, C Scheme, JAIPUR 302005	6 98 32
117/418 B Sarvodaya Nagar, KANPUR 208005	4 72 92
Patliputra Industrial Estate, PATNA 800013	6 23 05
Hantex Bldg (2nd Floor), Rly Station Road, TRIVANDRUM 695001	32 27

Inspection Office (With Sale Point):

Institution of Engineers (India) Building 1332 Shivaji Nagar PUNE 411005	5 24 35

*Sales Office in Bombay is at Novelty Chambers, Grant Road, Bombay 400007	[illegible] 65 2
†Sales Office in Calcutta is at 5 Chowringhee Approach, P. O Princep Street, Calcutta 700072	27 68 0

Printed at [illegible], New Delhi, India